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PATENT  
177079-00162

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicants : Sears et al.  
Serial No. : 10/616,045  
Filed : July 8, 2003  
For : **ALKYLENE OXIDE MODIFIED HARDWOOD  
CELLULOSE**  
Group Art Unit : 1623  
Examiner : N/A

919 Third Avenue  
New York, New York 10022

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Date of Deposit October 20, 2003

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**LETTER OF TRANSMITTAL**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

Enclosed for filing in the above-identified application is a Declaration under 37

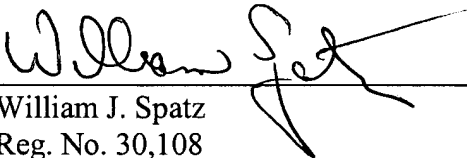
C.F.R. 1.132.

While no fees are believed due for this filing, the Commissioner is authorized to charge any fees or credit any overpayment to Deposit Account No. 50-0540. A duplicate copy of this letter of transmittal is enclosed.

Respectfully submitted,

KRAMER LEVIN NAFTALIS & FRANKEL LLP  
Attorneys for Applicants

By:

  
\_\_\_\_\_  
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**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of: Sears et al.

Serial No.: 10/616,045

Examiner: Not yet assigned

Filed: July 8, 2003

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*CONRAD DELACRUZ*

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**DECLARATION UNDER 37 C.F.R. 1.132**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, **Karl D. Sears**, declare and state:

1. I am one of the inventors of the referenced patent application.
2. I have received the following degrees: BA in Chemistry, University of Iowa (1963), and a Ph.D. in Organic Chemistry, University of Washington (1968).
3. I have worked as a scientist in the pulp and paper industry for over 35 years, and currently employed as the Principal Scientist at the Performance Fibers Research Center of the assignee of this patent application.
4. I am familiar with the subject matter disclosed and claimed in the referenced

patent application and with the contents of the Office Action mailed on April 8, 2003 in its parent patent application, Serial No. 09/943,650.

5. I am also the same Karl D. Sears who is the sole inventor of U.S. Patent No. 4,399,275 (hereinafter "*Sears 1*") cited in the Office Action referred to in Paragraph 4 above. I am fully familiar with the subject matter of *Sears 1*.

6. In patent application Serial No. 09/943,650, the Examiner relied on *Sears 1* to conclude that the invention of the present application is unpatentable under 35 U.S.C. § 103. Specifically, the Examiner found that it "would have been obvious to one having ordinary skill in the art, at the time the claimed invention was made, to have used the process of [*Sears 1*], and to make slight preferential adjustments in the type of cellulosic material used, and consequently, in the secondary reaction parameters like the temperature and the % by weight of the reagents, based on need, cost, availability and/or convenience of use. In view of the foregoing, the Examiner suggested that the applicant provide a "true side-by-side comparison of his parameters to [*Sears 1*] parameters, which consists of one common variable (such as temperature) and which indicates a showing of unexpected results in terms of the triacetate haze."

7. The Examiner of U.S. Application Serial No. 09/943,650 was incorrect in concluding that the examples contained in *Sears 1* do not enable a fair comparison of the results achieved by *Sears 1* with the presently claimed process, and in concluding that the claimed process is obvious in view of *Sears 1*. As explained herein, the examples of the present application demonstrate a process which has significant and surprising advantages over the prior art.

8. The presently claimed process is directed to a method for making cellulose acetate which is useful in commercial applications having stringent haze requirements. Consequently,

the process steps cannot destroy cellulose in the cellulosic material utilized in the process, and must yield cellulose acetate product having an intrinsic viscosity in the prescribed range for commercial applications (about 1.4 dl/gm to 1.7 dl/gm). Further, it must do the foregoing at commercially viable conditions and with a commercially viable yield. The claimed acetylation reaction uses an acid catalyst and extending the reaction time and/or raising the reaction temperature causes increased consumption of cellulose by the acid catalyst. Accordingly, the acetylation reaction temperature and the acetylation reaction time must be controlled so that 1) the reaction initiates; 2) the reaction proceeds to completion in a commercially reasonable time; and 3) the intrinsic viscosity of the cellulose acetate product falls within the prescribed range. Further, it is the standard to run an acetylation reaction for 170-190 minutes. In the chemical process industry, as in all industry, "time is money." Reaction conditions which require that an acetylation reaction run for significantly more than 190 minutes are disadvantageous.

9. In the case of the softwood pulp utilized in Sears 1, the acetylation reaction temperature had to be lowered to 19 °C in Example 6 to slow the reaction to a point where it could be controlled. The cellulosic material modified with 20% alkylene oxide was so reactive that at higher acetylation temperatures (in the range of 30 °C), the reaction would proceed so quickly that the exothermic heat of reaction would cause the temperature of the reactor contents to increase rapidly. At elevated temperature the acid catalyst consumes the cellulosic material generating additional heat, preventing the formation of cellulose acetate and/or yielding relatively small amounts of cellulose acetate having short cellulose chains which are characterized by an intrinsic viscosity which is below the prescribed range. Further, even at 19 °C, the reaction had to be terminated at a time when the intrinsic viscosity of the cellulose acetate product was in the prescribed range. For this reason, the acetylation reaction of

Example 6 of Sears 1 was terminated at 154 minutes. Extending the reaction time materially longer or significantly raising the reaction temperature would have resulted in the acid catalyst consuming more cellulose so that the intrinsic viscosity of any cellulose acetate product would be outside the acceptable range and decrease the product yield. In the present invention, the acetylation temperature of 32 °C was selected for Example 1 because that was the temperature necessary to get the alkylation reaction to initiate with the less reactive alkylene oxide modified hardwood cellulosic material and proceed to substantial completion within the industry standard time frame for this reaction, i.e., 170-190 minutes (see Sears 1, col. 6, lines 51-53). If the alkylation reaction were to be conducted at 19 °C, it would have proceeded, if at all, far more slowly and would not have progressed to an acceptable degree within 190 minutes.

10. The Examiner of the parent Application in the Official Action mailed April 8, 2003 took the position that “Although [Sears 1] uses a greater percentage of alkylene oxide (20% PO compared to 10% PO) to produce a cellulose acetate with approximately the same triacetate haze (6.7 compared to 6.5), this does not indicate that the use of a smaller PO percent, (like 10% PO) by [Sears 1] would not give cellulose acetate with a triacetate haze equal to or lower than 6.5% (i.e., better than applicant’s) at the acetylation temperature of 19 °C. Thus, for the same PO percent treatment [Sears 1] may obtain better triacetate haze at acetylation temperature of 19 °C as compared to applicant’s acetylation temperature of 32 °C”. However, Sears 1 does contain data which would enable those skilled in the art to determine the results of the experiment suggested to the Examiner. Specifically, Example No. 7 of Sears 1 subjected softwood cellulosic material which had been treated with 15% propylene oxide to an acetylation reaction at 23 °C and achieved a pulp having a triacetate haze level of 11.1. Further, a control pulp which was not treated with propylene oxide was acetylated at 28 °C and yielded cellulose

acetate having a triacetate haze value of 84.7. The results of these 3 examples are summarized below.

Sears 1 Example	Example 6	Example 7	Example 9
Propylene oxide added	20%	15%	0%
Percentage – $\text{OC}_3\text{H}_6\text{OH}$	4.4	3.6	0
Esterification Temp.	19 °C	23 °C	28 °C
Triacetate Haze	6.7	11.1	84.7

11. Clearly reducing the propylene oxide concentration in the processing of softwood pulp adversely affects the triacetate haze value of the cellulose acetate produced and eliminating the alkylene oxide treatment altogether results in cellulose acetate having an extraordinarily high triacetate haze level (84.7). It is evident to me that reducing the alkylene oxide concentration in Sears 1 from the 15% of Example 7 to 10%, as suggested by the Examiner of our parent application would result in cellulose acetate having a triacetate haze level considerably higher than the 11.1 value achieved with 15% PO. The minor difference in the esterification temperature in Example 6 (19 °C) and in Example 7 (23 °C) would not affect the triacetate haze value of the cellulose acetate produced and in any event was necessary since the 15% alkylene oxide treated pulp of Example 7 was less reactive than the 20% alkylene oxide treated pulp of Example 6. For this reason, it was necessary to slightly raise the esterification temperature in Example 7 and to continue the reaction for a longer time (154 minutes in Example 6 vs. 237 minutes in Example 7) to enable the reaction to proceed to substantial completion. The suggestion that reaction temperatures and times be changed ignores that meaningful results require that the desired reaction initiate and proceed to substantial completion, in a commercially reasonable time.



12. For the reasons described above, side-by-side comparisons, for example, of 10% PO modified hardwood and softwood cellulosic materials acetylated at 19 °C for like reaction times (say 170 to 190 minutes) would not present a fair comparison. The hardwood cellulosic material treated with 10% PO would not be reactive at 19 °C and the reaction would not proceed to a meaningful degree in 170 minutes. Hence there would be little or no cellulose acetate produced. On the other hand, the 19 °C reaction temperature and 170 minute reaction time would be favorable for the 10% PO modified softwood material, but would yield cellulose acetate having a triacetate haze materially higher than the 11.1 reported for the 15% PO material in Example 7 of Sears 1. If the same experiment were to be run at 32 °C, the reaction of the softwood cellulosic material would proceed so rapidly that the cellulose would be consumed by the acid catalyst yielding little if any cellulose acetate and the product would have an unacceptably low intrinsic viscosity. However, at 32 °C the reaction conditions would be favorable for the less reactive hardwood cellulosic material, so that cellulose acetate having substantially the specifications of Example 1 of the application would be produced.

13. In view of the foregoing, in my opinion as an expert in the field of cellulosic materials, with regard to the production of cellulose acetate the most meaningful comparison of the present invention with Sears 1 is presented by Example 6 of Sears 1 and Example 1 of the present application, as set forth below.

	Softwood Cellulosic Material Example 6 of Sears 1	Hardwood Cellulosic Material Example 1 of Present Appl.
PO Treatment	20%	10%
Propylene Oxide %	3.6	2.9
Acetylation Temperature	19 °C	32 °C
Reaction Time	154 minutes	180 minutes
Triacetate Haze	6.7	6.5



Both Examples were conducted at commercially reasonable conditions and proceeded to substantial completion in a commercially reasonable time. Notably, the use of hardwood cellulosic material offers the following significant and unexpected advantages over softwood:

- 1) it achieves a desirable 6.5 triacetate haze with only 10% PO;
- 2) the reaction could be conducted at 32 °C which did not require special cooling devices; and
- 3) the reaction proceeds to completion in a commercially reasonable 180 minutes.

14. As one of at least ordinary skill in the art, prior to the instant invention I would not have expected hardwood cellulosic material to have advantages over softwood material. Further, to my knowledge the invention of my original patent (Sears 1) was never put into commercial use because the trade considered the required low reaction temperature (i.e., approx. 19°C) and high alkylene oxide usage to be unacceptable. Since the present invention overcomes these problems, I expect that it will find commercial use.

I HEREBY DECLARE that all statements made herein to my knowledge are true and that all statements made on information and belief are believed to be true; and further, that the statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: October 20, 2003

By: Karl D. Sears  
Karl D. Sears